

## **Analysis of Heroin**

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### **1. Background**

Lysergic acid diethylamide (LSD), commonly referred to as “acid”, is a synthetic hallucinogen. LSD is manufactured from lysergic acid, which is found in ergot, a fungus that grows on rye and other grains. It is a colorless, odorless, and tasteless liquid. It comes in a variety of forms, but is always taken orally. LSD is most commonly found of small squares of paper called blotter (full sheet of paper are decorated with artwork or designs, perforated, then soaked in liquid solution and dried). Other forms include, tablets (microdots), gelatin squares (window panes), liquid, liquid sugar cubes and powder. Additionally, LSD has been embedded in candy such as “Gummy Worms,” “Sweet Tarts,” “Smarties,” and “Pez.”

### **2. Objective**

The objective of this SOP is to establish guidelines to be used for the analysis of a sample that may contain heroin.

### **3. Scope**

This SOP is to be used by the laboratory staff of the Division of Analytical Chemistry at William A. Hinton State Laboratory Institute in Boston, MA.

#### **4. Responsibility**

Chemists are responsible for acquiring glassware, preparing chemical reagents and standards, sample analysis, and reporting. Chemists also perform instrument calibrations, maintenance and troubleshooting, ordering of supplies and other necessary tasks related to this analysis.

Laboratory Supervisors ensure that chemists are following this SOP. They may perform the duties of the chemists and must review raw data and reports generated by chemists. The Supervisor may advise the chemists of alternative testing methods. They ensure that quality control measures are within acceptable limits and determine when corrective actions are needed. They coordinate proficiency testing (PT), reporting and distribution of PT results. They oversee sample results distribution to outside agencies.

Directors ensure that the SOP is being followed and reviewed on a regular basis. They provide approval of standard operating procedures and review quality control documentations.

#### **1. Related Documents**

Cole, Michael, "The Analysis of Controlled Substances," London: John Wiley & Sons Ltd., 2003  
Drug Enforcement Administration, "Basic Training Program for Forensic Drug Chemists," Drug Enforcement Administration.

Mills III, Terry et al, "Instrumental Data for Drug Analysis," 3<sup>rd</sup> ed., 6 vols., New York: CRC Press, 2006.

Moffat, A.C. et al, "Clarke's Isolation and Identification of Drugs," 2<sup>nd</sup> ed., London: The Pharmaceutical Press, 1986.

Moffat, A.C. et al. "Clarke's Analysis of Drugs and Poisons," 3<sup>rd</sup> ed., London: The Pharmaceutical Press, 2004.

Saferstein, Richard, "Forensic Science Handbook," New Jersey: Prentice Hall, 1988.

Scientific Working Group for the Analysis of Seized Drug Recommendation, 6<sup>th</sup> ed., "Part III A & B, Methods of Analysis/Sampling of Seized Drug for Qualitative Analysis," July 2011

#### **6. Definitions**

GC: Gas Chromatography

GC/MS: Gas Chromatography/Mass Spectrometry

#### **7. Supplies, Equipment & Reagents**

##### **Supplies**

Beakers

Spatula

Pasteur pipette

Volumetric flask

Weighing dish

Weighing paper

GC vials with Teflon caps

##### **Equipment**

Analytical Balance

Ultraviolet (UV) Lamp

GC with FID

**GC/MS**

**Reagents**

p-dimethylaminobenzaldehyde (p-DMAB)  
Lysergic acid diethylamide (LSD)  
Lysergic acid methylpropylamide (LAMPA)  
95% Ethanol  
Concentrated Hydrochloric Acid  
Methanol  
Cocaine Hydrochloride  
Codeine Phosphate  
Chloroform  
Sodium Bicarbonate  
Tartaric Acid  
Water

**8. Safety**

Due to the potential hazards, appropriate precautions should be taken as necessary. This includes, but is not limited to, the use of fume hoods, gloves, masks and safety glasses. Lab coats are to be worn at all times in the unit, unless performing administrative duties.

**9. Reagent Preparation**

**Cobalt Thiocyanate Reagent**

Dissolve 2.0g of cobalt thiocyanate in 100mL of deionized water. Mix the solution until completely dissolved.

**Marquis Reagent**

Dilute 10mL of 37% formaldehyde solution in 90mL of concentrated sulfuric acid. While stirring, slowly add the concentrated sulfuric acid to the formaldehyde solution. Allow the solution to cool completely.

**Froedhde's Reagent**

Dissolve 0.5g of sodium molybdate in 100mL of concentrated sulfuric acid. Mix the solution until completely dissolved.

**Mecke's Reagent**

Dissolve 1.0g of selenous acid in 100mL of concentrated sulfuric acid. Mix the solution until completely dissolved.

**2.8N Hydrochloric Acid Reagent**

Dilute 92.6mL of 12.1N hydrochloric acid in 400mL of deionized water. Mix the solution completely.

**Cocaine/Codeine Standard (QC Mix)**

Dissolve 10.0 mg of cocaine hydrochloride and 10.0mg of codeine phosphate and bring to volume with 10mL of methanol. Mix the solution until completely dissolved.

Heroin Standard

Dissolve 10mg of heroin hydrochloride in 10mL of methanol. Mix the solution until completely dissolved.

Quantitative Standards

**10. Procedure**

- A. Evidence Handling
- B. Document observations on the Drug Analysis Form noting the number, type (e.g. powder, crystalline, liquid or residual device) and marking of all items.
- C. Sampling Plan
  - i. Attempt to scrape or remove sample from the device and place onto weighing paper or
- D. Residues
  - i. Attempt to scrape or remove sample from the device and place onto weighing paper or boat.
  - ii. Or rinse the device containing the sample with 1-2ml of the chloroform and place the extract into a beaker.
  - iii. Transfer some of the sample or extract into a labeled residue vial for GC and GC/MS analysis. Cap and seal the vial tightly.
  - iv. Use the remaining sample or extract to perform the color test.
- E. Color Test
  - i. The color test consists of four reagents, which are cobalt thiocyanate, marquis, froehde's, and mecke's.
  - ii. Place a couple of drops of cobalt thiocyanate, marquis, froehde's, mecke's reagents into individual wells on a porcelain spot plate.
  - iii. Add a small amount of sample (1-2mg of powder or 1-2 drops of liquid) to each well.
  - iv. Note any color change or reaction. The results will be recorded on the Drug Analysis Form by documenting the actual color/s observed. Negative observations will be recorded by stating no reaction present or no color change.
- F. Interpretation
  - i. Marquis reagent: Formation of a purple color indicates the possible presence of heroin, other opiates, methocarbamol or guaifenesin.
  - ii. Froehde's reagent: Formation of a purple color indicates the possible presence of heroin and other opiates.
  - iii. Mecke's reagent: Formation of a green color indicates the possible presence of heroin and other opiates.
- G. Gas Chromatography (as necessary)
  - i. Place 1-2mg of powder into a labeled GC vial and then add 1.8mL of methanol.
  - ii. Initiate auto sampler sequence using the ROUTINE method running a blank solvent between each unknown sample and reference standard/s.
  - iii. Compare retention time of the each sample with the reference standard/s. Also check the chromatograph to determine if the sample needs to be diluted or concentrated.
  - iv. Positive GC analysis will be recorded on the Drug Analysis Form by the use of a plus (+). The result is considered positive when the retention time of the sample and the reference standard meet the laboratory criteria and are specified in the notes. Negative observations will be recorded by the use of a negative (-).
- H. Gas Chromatography/Mass Spectrometry

- i. Confirmatory analysis can be performed using the GC vial from the previous section (E).
- ii. Initiate auto sampler sequence using the DRUGS method running a blank solvent between each unknown sample and reference standard/s.
- iii. Compare retention time and ion spectra of the each sample with the reference standard/s (Heroin).
- iv. Document the date analyzed and results of the GC/MS onto the MS Tracking Sheet, Drug Analysis Form and Control Card.

**I. Quantitation**

**11. Documentation**

- A. All results will be documented on the Drug Analysis Form.
- B. All raw data will be generated and filed according to the laboratory policy.
- C. A certificate of analysis will be generated for each lab number which will document the results.

**12. Attachments**

**GC Method**

**GC/MS Method**